



PATENT  
ATTORNEY DOCKET NO. 0092/012001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Masaaki OYAMADA et al. Art Unit: 1762  
Application No.: 10/820,024 Examiner: Tsoy, E.  
Filing Date: April 8, 2004  
Title : CONDUCTIVE ELECTROLESSLY PLATED POWDER AND  
METHOD FOR MAKING SAME

Commissioner for Patents  
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DECLARATION UNDER 37 CFR 1.132

Sir:

I, Shinji Abe, do declare and state as follows:

I am one of the applicants of the above-identified application;

I have been employed by NIPPON CHEMICAL INDUSTRIAL CO., LTD for 10 years as a researcher in the field of conductive electroless plated powders;

I have read all of the Office Actions in the above-entitled application, and have read and am familiar with each of the references cited in the Office Actions by the Examiner; and

The following experiments were carried out by me or under my direct supervision and control and the results are true and correct to the best of my knowledge.

## I. Object

The following experiments relate to conductive electroless plated powders used in the present invention and Weber et al. (U.S. Patent No. 6,274,241).

## II. Brief Description of the Drawings

FIG. 1 is an optical microscope photograph (magnification of transmitted light: 500) showing a plating powder obtained in Experiment 1.

FIG. 2 is an optical microscope photograph (magnification of transmitted light: 500) showing a plating powder obtained in Experiment 2.

FIG. 3 is an optical microscope photograph (magnification of transmitted light: 500) showing a plating powder obtained in Experiment 3.

FIG. 4 is a reflection electron composition image with an electron microscope photograph (magnification: 5,000) showing the plating powder obtained in Experiment 2.

FIG. 5 is an optical microscope photograph (magnification of transmitted light: 500) showing a plating powder of Example 1 of the present invention.

### III. Experiments

#### Preparation of Catalyzation Particles

Catalyzation particles were prepared in accordance with Example 1 of the present invention.

Spherical silica with an average particle size of 12  $\mu\text{m}$  and an absolute specific gravity of 2.23 were used as the core particles. The spherical silica (40 g) was added to 400 ml of an aqueous conditioner solution (Cleaner Conditioner 231 manufactured by Shipley Corporation) while being stirred. The concentration of the aqueous conditioner solution was 40 ml/l. Stirring was continued for 30 minutes at a solution temperature of 60°C under ultrasonic irradiation to perform surface treatment and dispersion. The aqueous solution was filtered and the core particles were subjected to repulping-washing with water (in the so called "repulping washing", the core particles are re-slurried and washed with water) one time and formed into a 200 ml slurry. To the slurry was added 200 ml of an aqueous solution of tin chloride. The concentration of the aqueous solution was  $5 \times 10^{-3}$  moles/l. Stirring was performed at a normal temperature for 5 minutes to perform sensitization in which tin ions were allowed to adsorb to the surfaces of the core particles. The aqueous solution was then filtered and repulping-washing with water was

performed one more time. The core particles were formed into a 400 ml slurry and maintained at 60°C. While stirring the slurry under ultrasonic irradiation, 2 ml of an aqueous palladium chloride solution (0.11 moles/l) was added to the slurry. Stirring was continued for another 5 minutes to perform activation in which palladium ions were captured by the surfaces of the core particles. The aqueous solution was then filtered, and the core particles were subjected to repulping-washing with hot water one time and formed into a 200 ml slurry. The slurry was stirred under ultrasonic irradiation, and 20 ml of a mixed aqueous solution of dimethylamine borane (0.017 moles/l) and boric acid (0.16 moles/l) was added thereto. Stirring was performed at a normal temperature for 2 minutes under ultrasonic irradiation to reduce the palladium ions. The aqueous solution was filtered and the core particles were subjected to repulping-washing with water. The core particles were then dried.

#### Preparation of the Plating Bath

The plating bath was prepared as described in column 7, lines 26-40 of Weber et al.

The plating bath solution was composed of

49.5g  $\text{Na}_2\text{WO}_4$ ,

65.4g Na gluconate,

6.57g  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ,  
5.13g dimethylaminobrane,  
50mg thiourea,  
approx. 500 ml  $\text{H}_2\text{O}$ , and  
approx. 75 ml of a 25% ammonia solution.

The plating bath had a pH of 9, was heated to  $80^\circ\text{C}$  and stirred.

#### Electroless Plating Step

##### (1) Experiment 1

As soon as, 500 ml of the above plating bath was adjusted, 10 g of core particles that were obtained as described in the catalyzation particles producing step were quickly added to the plating bath.

After the completion of the addition of the core particles, stirring was continued while maintaining the temperature at  $80^\circ\text{C}$  for 1 hour. Then the plating bath solution was filtered and the particles were subjected to repulping-washing with water. Then, the particles were vacuum-dried at  $110^\circ\text{C}$ . The particles obtained are shown in Fig. 1.

##### (2) Experiment 2

10 g of core particles that were obtained as described

in the catalyzation particles producing step were added in 100 ml  $H_2O$  and formed a slurry solution. Thereafter, its temperature was increased to  $80^{\circ}C$ . As soon as, 500 ml of the above plating bath solution was adjusted, it was added in the slurry solution quickly. After the completion of the addition of the plating bath solution, stirring was continued while maintaining the temperature at  $80^{\circ}C$  for 1 hour.

Then the plating bath was filtered and the particles were subjected to repulping-washing with water. Then, the particles were vacuum-dried at  $110^{\circ}C$ . The particles obtained are shown in Figs. 2 and 4.

### (3) Experiment 3

10 g of core particles that were obtained as described in the catalyzation particles producing step were added in 100 ml  $H_2O$  and formed a slurry solution. Thereafter, its temperature was increased to  $80^{\circ}C$ . As soon as, 500 ml of the above plating bath was adjusted, it was added in the slurry solution at an addition rate of 7.0 ml/min in accordance with the rate described in Example 1 of the present invention.

After the completion of the addition of the slurry solution, stirring was continued while maintaining the

temperature at 80°C for 1 hour.

Then the plating bath solution was filtered and the particles were subjected to repulping-washing with water. Then, the particles were vacuum-dried at 110°C. The particles obtained are shown in Fig. 3.

#### IV. Result and Discussion

The result of Experiment 1:

After adding the metal solution to the core particles, no metal reaction occurred.

The result of Experiment 2:

After adding the metal solution to the slurry solution, the pH was changed and this caused the metal solution to decompose.

The result of Experiment 3:

When slowly adding the metal solution, no metal reaction occurred. After adding the metal solution, the pH was changed and this caused the metal solution to decompose.

Optical microscope photographs of the plating powders produced in Experiments 1 to 3 are shown in Figs. 1 to 3.

Since nickel metal was not disposed on the surface of the core particles in Experiments 1 to 3, the light from the optical microscope went through the particles. Therefore, since almost all of the plating powders shown in

Figs. 1 to 3 are not completely coated, they are shown as being transparent in these figures. We also confirmed that decomposed fine nickel metal powders existed around the core particles.

Experiment 2 shown in Fig. 2 seemed to have the most coating, so we obtained a scanning electron microscope photograph of the plating powders produced in Experiment 2 and this is shown in Fig. 4. We also confirmed that there were no columnar structures of the nickel metal on the surface of the core particles. In Fig. 4, the surface of the core particles are not coated with Ni metal but rather it is shown with some randomly scattered Ni metal particles thereon.

On the other hand, as shown in Fig. 5, since nickel metal was uniformly deposited on the surface of the core particles in the present invention, the light from the optical microscope did not go through the coated particles. Therefore, all of the plating powders in Fig. 5 are shown in solid black (not transparent). As further discussed in the present invention in connection with Example 1, this coating includes the columnar structure shown in Fig. 1 of the present invention.

## V. Conclusion

As discussed above and shown in Figs. 1 to 4, the each plating powders produced by Experiments 1 to 3 did not have a uniform and continuous metal film on the surface of the core particle.

We also confirmed that these core particles did not have any columnar structure on the surface of their core particles.

I further declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: March 23, 2007

Shinji Abe  
Shinji ABE

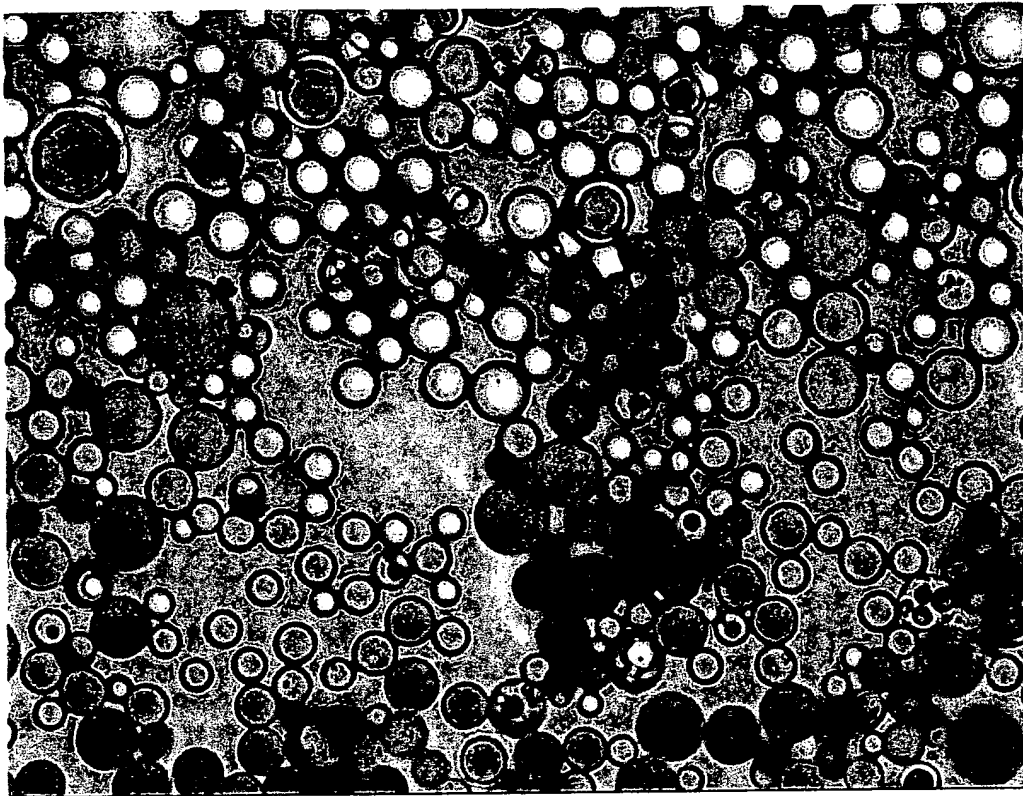


FIG.1

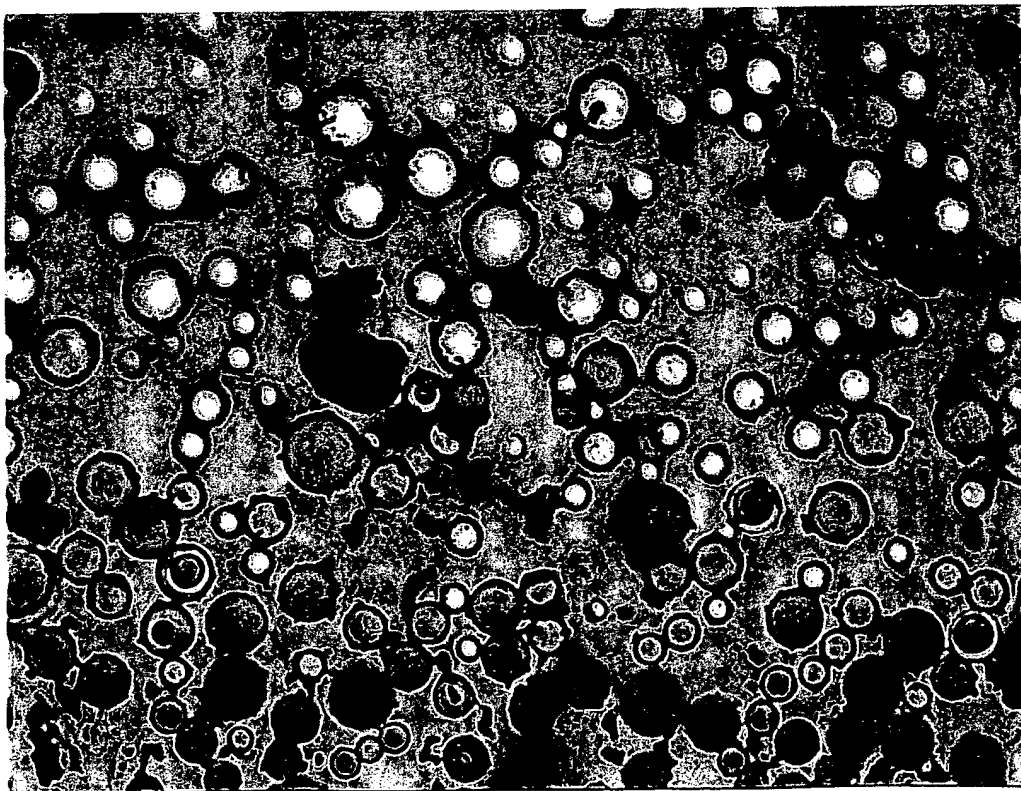


FIG.2

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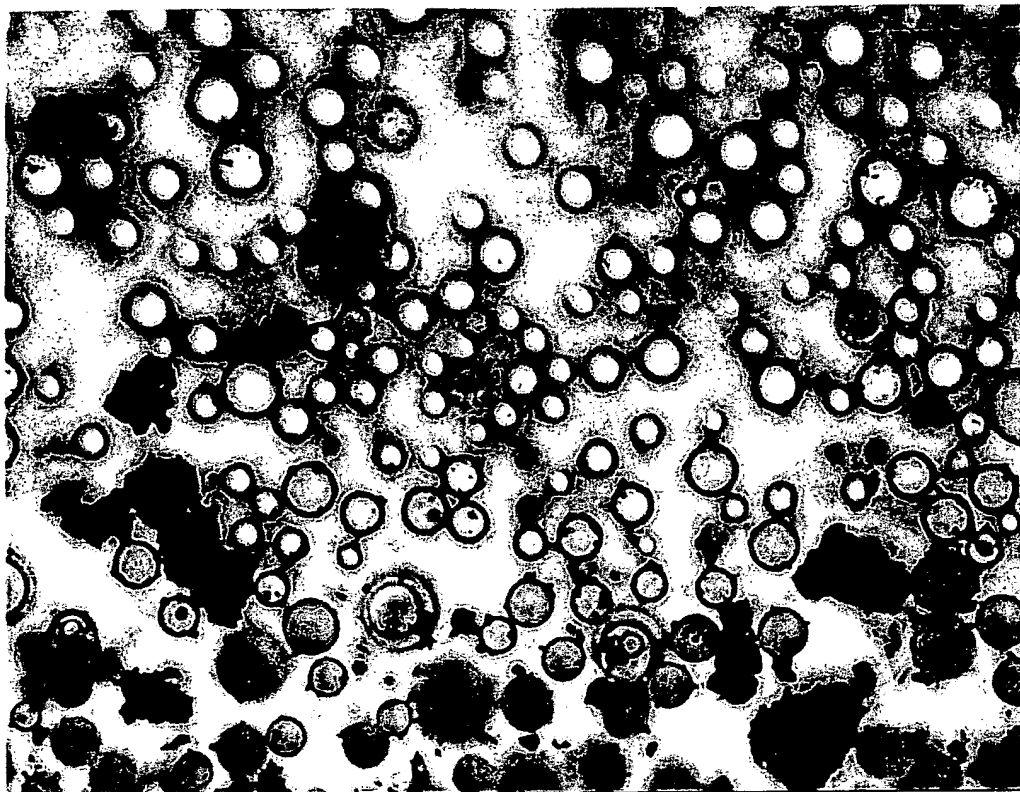


FIG.3

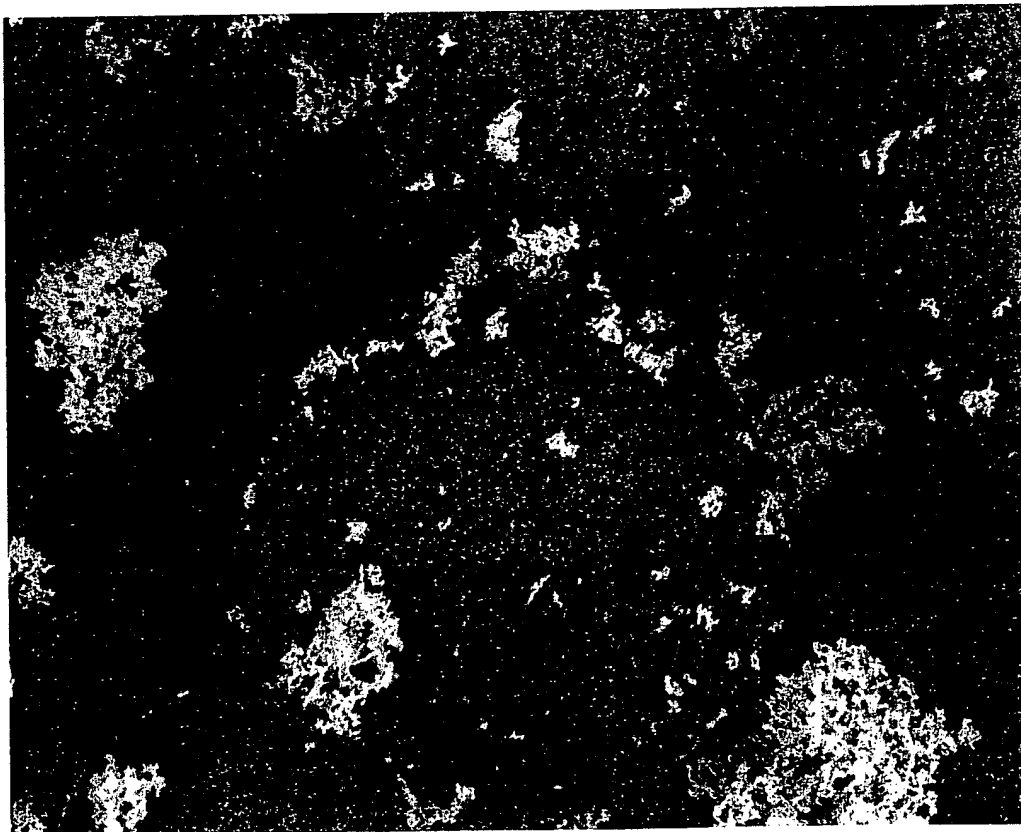


FIG.4

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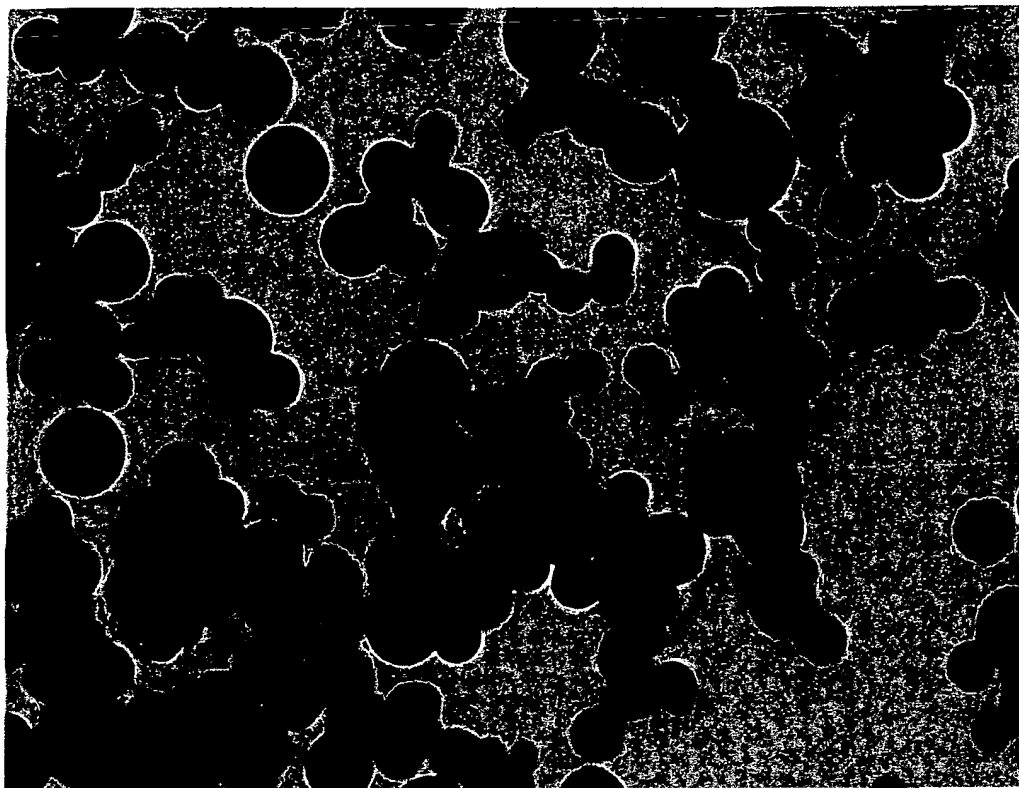


FIG.5

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